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Cardiff Road
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1. Your reference — MPD315/GB/RGMS

2. Patent application number *(The Patent Office will fill in this part)* — 10 JUN 1999 — **9913408.2**

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

   ALBRIGHT & WILSON UK LIMITED
   210-222 HAGLEY ROAD WEST
   OLDBURY
   WEST MIDLANDS B68 0NN

   Patents ADP number *(if you know it)* 6804264002 Rdas

   If the applicant is a corporate body, give the country/state of its incorporation — ENGLAND

4. Title of the invention

   PERSONAL CARE FORMULATIONS

5. Name of your agent *(if you have one)* — R G M SAVIDGE

   "Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

   ALBRIGHT & WILSON UK LIMITED
   PATENTS DEPARTMENT
   210-222 HAGLEY ROAD WEST
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   Patents ADP number *(if you know it)* 7383219001 Rdas

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Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

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11. I/We request the grant of a patent on the basis of this application.

Signature
R G M SAVIDGE – By Power of Attorney

Date 09.06.99

12. Name and daytime telephone number of person to contact in the United Kingdom

MR R G M SAVIDGE
0121 420 5430

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PERSONAL CARE FORMULATIONS

The present invention relates to shampoo or cleaning compositions suitable for personal care applications in the form of I_1 mesophase systems containing dispersed oil.

Dispersing oil in aqueous shampoo and body wash formulations has presented problems. To prevent the oil phase separating it must either be: (A) emulsified which involves dispersing the oil as colloidal single droplets; (B) microemulsified which involves forming a micellar solution with oil incorporated into surfactant micelles; (C) suspended in a structured surfactant system which typically comprises a dispersion of a surfactant mesophase in aqueous electrolyte; or (D) incorporated into a water soluble solid, pasty or gelatinous composition.

With the exception of microemulsions which are clear, thermodynamically stable, micellar solutions, the foregoing systems are necessarily opaque and contain the oil dispersed in a relatively coarse form, which does not deposit satisfactorily on skin or hair.

However microemulsions are difficult to formulate using the surfactants which are most effective in body wash and other personal care formulations and contain relatively low concentrations of surfactant.

We have now discovered that oil may be stably incorporated into the structure of an I_1 phase to form a clear gel-like composition which contains higher concentrations of surfactant and oil than conventional microemulsions, but, which dissolves in water to form a microemulsion. The novel oil-in-I_1 compositions also form microemulsions on heating.

Surfactants are known to form mesophases or liquid crystal phases at concentrations above approximately 30% by weight based on the weight of water and surfactant. Mesophases are phases which exhibit a degree of order intermediate between typical liquids and solids. Generally mesophases combine long range order associated with crystals, with fast molecular motion common to liquids.

The formation of detergent mesophases is well documented. Different surfactants and surfactant mixtures differ widely in their ability to form the numerous different mesophases, and in respect of the conditions of concentration and temperature at which they are formed. For a typical surfactant of the type normally used in cleaning products the following mesophases are usually observed. The concentrations given are illustrative only and may vary considerably from one surfactant or surfactant mixture to the next.

Below approximately 30% surfactant an isotropic L_1 phase is formed (with micelles of surfactant in water). Above 30% surfactant many detergents form a M phase which is not normally used in personal care applications since it dos not show suitable flow characteristics and is difficult to dissolve or disperse in water. Above the concentrations required to form an M phase but usually at concentrations of less than 80% active surfactant, i.e. 60%-80% a G-phase is formed. At concentrations higher than those required to form a G-phase, i.e. typically greater than 80% active surfactant, most surfactants form a hydrated solid, and some, especially non-ionic surfactants form a liquid phase containing dispersed micelle sized droplets of water - an inverted micellar solution known as an L_2 phase. L_2 detergent systems do not disperse readily in water and have a tendency to form undesirable gels, e.g. M phases, on dilution.

Some surfactants form viscous isotropic or VI phases. These are immobile phases usually with a vitreous appearance, and have been relatively little studied compared to the other phases discussed above. They have been virtually ignored in the context of formulating cleaning compositions because most of the surfactants and surfactant systems which are commonly used in cleaning compositions do not form VI phases, at least at normal temperatures, or form them only within narrow concentration ranges and because their known properties as immobile gels has deterred formulators from investigating them. They are recognised as being the most viscous of the lyotropic mesophases.

The different surfactant phases can be recognised by a combination of appearance, rheology, textures under the microscope, electron microscopy and x-ray diffraction or neutron scattering. A detailed description, with illustrations, of the difference textures observable using a polarising microscope, is to be found in the paper by Rosevear JAOCS Vol 31, p628.

The following terms may require explanation or definition:

The "hydrophilic: lipophilic balance", or "HLB" value is used as a measure of the relative affinities of the surfactants for water and oil respectively and correlates with their effectiveness as emulsifiers. HLB value can easily be calculated for alcohol ethoxylates since it is one fifth of the weight percent of ethylene oxide based on the total mole weight. Other surfactants can be assigned equivalent values by applying more complicated formulae or by measuring their relative affinity for water and oil. An HLB value of 20 represents a completely water soluble oil insoluble surfactant, while an HLB value of 0 represents a completely oil soluble and water insoluble surfactant.

"Optically isotropic" surfactant phases do not normally tend to rotate the plane of polarisation of plane polarised light. If a drop of sample is placed between two sheets of optically plane polarising material whose planes are at right angles, and light is shone on to one sheet, optically isotropic surfactant samples do not appear substantially brighter than their surrounding when viewed through the other sheet. Optically anisotropic materials appear substantially brighter. Optically anisotropic mesophases typically show characteristic textures when viewed through a microscope between crossed polarisers, whereas optically isotropic phases usually show a featureless continuum.

"Newtonian liquids" have a viscosity which remains constant at different shear rates. For the purpose of this specification, liquids are considered Newtonian if the viscosity does not vary substantially at shear rates up to 1000 sec^{-1}.

"Lamellar" phases are phases which comprise a plurality of bilayers of surfactant arranged in parallel and separated by liquid medium. They include both solid phases and the typical form of the liquid crystal G-phase. G-phases are typically pourable, non-Newtonian, anisotropic products. They are typically viscous-looking, opalescent materials with a characteristic "smeary" appearance on flowing. They form characteristic texture under the polarising microscope and freeze fractured samples have a lamellar appearance under the electron microscope. X-ray diffraction or neutron scattering similarly reveal a lamellar structure, with a principal peak typically between 4 and 10nm, usually 5 to 6nm. Higher order peaks, when present occur at double or higher integral multiples of the Q value of the principal peak. Q is the momentum transfer vector and is related, in the case of lamellar phases, to the repeat spacing d by the equation $Q=\frac{2n}{d}$ [pi] where n is the order of the peak.

G-phases, however, can exist in several different forms, including domains of parallel sheets which constitute the bulk of the typical G-phases described above and spherulites formed from a number of concentric spheroidal shells, each of which is a bilayer of surfactant. In this specification the term "lamellar" will be reserved for compositions which are at least partly of the former type. Opaque compositions at least predominantly of the latter type in which the continuous phase is a substantially isotropic solution containing dispersed spherulites are referred to herein as "G-phase compositions". G-phases are sometimes referred to in the literature as $L_{(alpha)}$ phases.

L_1-phases are mobile, optically isotropic, and typically Newtonian liquids which show no texture under the polarising microscope. Electron microscopy is capable of resolving the texture of such phases only at very high magnifications, and X-ray or neutron scattering normally gives only a single broad peak typical of a liquid structure, at very small angles close to the reference beam. The viscosity of an L_1-phase is usually low, but may rise significantly as the concentration approaches the upper phase boundary.

"M-phases" are typically immobile, anisotropic products resembling low melting point waxes. They give characteristic textures under the polarising microscope, and a hexagonal diffraction pattern by X-ray or neutron diffraction which comprises a major peak, usually at values corresponding to a repeat spacing between 4 and 10nm, and sometimes higher order peaks, the first at a Q-value which is $3^{0.5}$ times the Q-value of the principal peak and the next double the Q-value of the principal peak. M-phases are sometimes referred to in the literature as H-phases.

The viscous isotropic or "VI" phases are typically immobile, non-Newtonian, optically isotropic and are typically transparent, at least when pure. VI phases have a cubic symmetrical diffraction pattern, under X-ray diffraction or neutron scattering, with a principal peak and higher order peaks at $2^{0.5}$ and $3^{0.5}$ times the Q-value of the principal peak.

These cubic liquid crystalline phases are sometimes observed immediately following the micellar phase at ambient temperature as the concentration of surfactant is increased. It has been proposed that such VI phases, sometimes referred to as I_1 phase, may arise from the packing of micelles (probably spherical) in a cubic lattice. At ambient temperature a further increase in surfactant concentration usually results in hexagonal phase (M_1), which may be followed by a lamellar phase (G). I_1 phases, when they occur, are usually only observed over a narrow range of concentrations, typically just above those at which the L_1-phase is formed. The location of such VI phases in a phase diagram suggests that the phase is built up of small closed surfactant aggregates in a water continuum.

An inverse form of the I_1 phase (the I_2 phase) has also been reported, possibly between the inverse hexagonal (M_2) and L_2 phases. It consists of a surfactant continuum containing a cubic array of inverted micelles. An alternative form of the VI phase called the V_1 phase has been observed at concentrations between the M and G phases and may comprise a bicontinuous system. This may exhibit an even higher viscosity than the I_1. An inverse phase, the V_2 phase between the G and M_2 phases has also been postulated.

VI phases are typically examples of "ringing gels". When a jar or beaker containing such a phase is sharply struck, a distinctive vibration can be felt in the composition.

The I_1/L_1 transition temperature will be referred to herein as the melting point of the I_1 phase for convenience, although it is not strictly speaking the melting point since the VI phases are not solids.

All references herein to the formation or existence of specific phases or structures are to be construed, unless the context requires otherwise, as references to their formation or existence at 20°C..

Hexagonal gels (M-phase) have been referred to in the prior art as cleaning compositions, e.g. GB 2 179 055, EP I 153 837 and colloidal gels formed with gelling agents such as synthetic polymers or gelatin have also been suggested, e.g. US 4 465 663.

However these compositions cannot be readily dissolved in water to form microemulsions. They are moreover usually opaque and of an unattractive appearance and often require the presence of solvents such as glycols which add to the cost and are environmentally undesirable.

The use of a type of ringing gel to suspend oil for cosmetic or pharmaceutical applications was described in US 4 026 818 but the formulation requires the presence of hydroxylic solvents and utilises a surfactant system which is unsuitable for shampoo applications. EP O 598 335 describes the use of various cubic phases including I_1 phases as laundry prespotters and for other cleaning formulations. If does not suggest how such phases could be used to suspend oil or form microemulsions. Normally attempts to suspend oil in surfactant mesophases result in coarse droplets of oil being suspended in the aqueous phase of a structured surfactant.

Our invention provides a concentrated personal cleansing composition comprising at least 20% water, 10 to 40% total surfactant and 2 to 40% of a mineral, glyceride, terpene or silicone oil wherein said surfactant comprises (A) an oil soluble surfactant having an HLB of from 2 to 10 in a proportion of from 8:1 to 1:5 based on the weight of oil and (B) a hydrophilic surfactant having an HLB greater than 11, in a weight proportion of from 1:1 to 1:30 based on the weight of (A), said surfactant water and oil being present in proportions adapted to form an I_1 phase having an I_1/L_1 transition temperature greater than 25°C.

The surfactants are preferably selected to provide an I_1 phase over a comparatively broad surfactant concentration range e.g. more than ± 5% or greater, which range typically lies above 15% by weight total surfactant based on the weight of the composition e.g. between 20% and 40% by weight surfactant usually between 25% and 60%.
The surfactants are preferably selected to provide an I_1 phase which melts above 30°C e.g. above 35°C, most preferably above 40°C. Preferably the I_1 phase melts at a temperature substantially below 100°C, e.g. below 90°C, more preferably below 80°C, most preferably below 70°C, especially below 60°C, typically below 55°C, usually below 50°C.

The surfactant mixture preferably has a mean HLB based on the molar proportions of the components between 10 and 15 e.g. 11 to 14. The surfactants preferably comprise non-ionic surfactants such as ethoxylated alcohols. It has been found that highly ethoxylated fatty alcohols, e.g. more than 10 EO groups, preferably more than 15 EO groups, especially 18 to 50 EO groups form I_1 phases particularly readily.

Other non-ionic surfactants which may be present include:

alkyl phenol ethoxylates, fatty acid ethoxylates, fatty acid monoalkylolamide ethoxylates, fatty alcohol propoxylates, fatty anime alkoxylates and fatty acid glyceryl ester ethoxylates. Other non-ionic compounds suitable for inclusion in compositions of the present invention include mixed ethylene oxide propylene oxide alkoxylates, low relative molecular mass polyethylene glycols e.g. PEG600 and PEG200, ethylene glycol monoesters, amine oxides and alkyl polyglycosides, alkyl sugar esters including alkyl sucrose esters and alkyl oligosaccharide ester, alkyl capped polyvinyl alcohol and alkyl capped polyvinyl pyrrolidone.

Compositions of the invention may also comprise anionic surfactants, in addition to or instead of non-ionic surfactants. Anionic surfactant may comprise a C_{10-20} alkyl benzene sulphonate or an alkyl ether sulphate which is preferably the product obtained by ethoxylating a natural fatty or synthetic C_{10-20} e.g. a C_{12-14} alcohol with from 1 to 20, preferably 2 to 10 e.g. 3 to 4 ethyleneoxy groups, optionally stripping any unreacted alcohol, reacting the ethoxylated product with a sulphating agent and neutralising the resulting alkyl ether sulphuric acid with a base. The term also includes alkyl glyceryl sulphates, and random or block copolymerised alkyl ethoxy/propoxy sulphates.

The anionic surfactant may also comprise, for example, C_{10-20} e.g. C_{12-18} alkyl sulphate.

The surfactant may comprise a C_{8-20} e.g. C_{10-20} aliphatic soap. The soap may be saturated or unsaturated, straight or branched chain.

Preferred examples include dodecanoates, myristates, stearates, oleates, linoleates, linoleates and palmitates and coconut and tallow soaps.

The surfactant may include other anionic surfactants, such as olefin sulphonates, paraffin sulphonates, taurides, isethionates, ether sulphonates, ether carboxylates, aliphatic ester sulphonates e.g. alkyl glyceryl sulphonates, sulphosuccinates or sulphosuccinamates.

The cation of any anionic surfactant is typically sodium but may alternatively be potassium, lithium, calcium, magnesium, ammonium, or an alkyl ammonium having up to 6 aliphatic carbon atoms including isopropyl ammonium, monoethanol ammonium, diethanol ammonium, and triethanol ammonium.

Ammonium and ethanol ammonium salts are generally more soluble than the sodium salts. Mixtures of the above cations may be used.

The composition may contain amphoteric surfactants such as betaines sulphobetaines, amido betaines or imidazoline betaines.

The I_1 phase may be conveniently prepared by mixing the oil and oil soluble surfactant and adding sufficient water to the water soluble surfactant to maintain a lamellar phase. The oil and oil soluble surfactant may be stirred into the lamellar composition at elevated temperature, above the melting point of the desired I_1 phase. The composition is then diluted with hot water until a microemulsion is formed and then cooled to solidify it into the I_1 phase.

The oil is preferably a mineral oil (e.g. a low molecular weight petroleum ether) or a fatty glyceride, a terpene oil such as limonene or a silicone oil. Mixtures of oils may be used. Particularly preferred are vegetable oils such as coconut, evening primrose, groundnut, meadow foam, apricot kernel, peach kernel, avocado, jojoba and olive oil. Oil soluble cosmetic or topical pharmaceutical ingredients may be dissolve in the oil including antiseptics, styptics, antidandruff agents such as zinc omadine (zinc pyrithione) and selenium disulphide, proteins, emollients such as lanolin, isopropyl myristate, glyceryl isostearate or propylene glycol distearate, dyes, perfumes and waxes. Water insoluble particulate solids including exfoliants such as talc, clays, polymer beads, sawdust, silica, seeds, ground nutshells and dicalcium phosphate, pearlisers such as mica or glycerol or ethylene glycol mono- or di-stearate, glitter additives and sunscreens such as titanium dioxide may be dispersed in the hot microemulsion prior to cooling. Porous particles (so called micro-sponges) containing absorbed active ingredients or gelatin or other microcapsules may be suspended. Other active ingredients which may be suspended include insect repellants and topical pharmaceutical preparations, e.g. preparations for treatment of acne, fungicides for athlete's foot or ringworm or antiseptics or antihistamines. Pigments, such as the iron oxides, may also be added.

Electrolytes tend to break I_1 phase structure and are preferably present in concentrations below 10% based on total weight of the compositions, more preferably below 5%, e.g. 0 to 3%, most preferably 0 to 1%. Generally we prefer that electrolyte be substantially absent. Adventitious chloride or sulphate present as impurities in the surfactant can be tolerated. Small amounts of builder such as citrates, pyrophosphates, polyphosphates may optionally be included.

Water soluble solvents are generally undesirable and are not required to form stable I_1 structures according to the invention. We therefore prefer that they should be substantially absent. Although small amounts of, for example, ethanol or propanol may sometimes be desired for special purposes, they are preferably present in amounts less than 5% by weight, more preferably less than 3% by weight, most preferably less than 2% by weight, e.g. less than 1% by weight.

The composition may optionally contain hydrotropes such as sodium lower alkyl benzene sulphonate e.g. sodium toluene, xylene or cumene sulphonate or urea, however these are not generally necessary and are not generally preferred. We prefer that these should be present in quantities less than 5% by weight, more preferably less than 4%, especially less than 2% e.g. 0 to 1%. They may be useful occasionally to avoid haziness of the gel.

The total amount of water is preferably from 25 to 60% by weight of the composition, more preferably 30 to 50%, e.g. 35 to 50%. The total weight percentage of surfactant based on the weight of the composition is preferably from 15 to 35%, e.g. 20 to 30%. The proportion of oil is preferably greater than 5%, more preferably greater than 8%, e.g. 10 to 30%, especially 15 to 25% by weight based on the weight of the composition. The oil soluble surfactant is preferably present in a proportion of more than 1:5 based on the weight of oil, more preferably from 1:2 to 5:1. The oil soluble surfactant preferably has an HLB of from 3 to 9 e.g. 4 to 8.

The weight ratio of water soluble surfactant to oil soluble surfactant is preferably 1:1 to 30:1, more preferably 2:1 to 20:1, typically 3:1 to 15:1, e.g. 4:1 to 10:1. The water soluble surfactant preferably has an HLB greater than 12, more preferably greater than 13, especially 14 to 19.

The product may be cast into shaped bodies or formed into particles or granules, e.g. by spray cooling a hot solution of the L_1 phase formed on melting the composition.

The composition may be converted into a microemulsion phase by addition of water, by heating above the melting point or by adding electrolyte such as salt and the invention includes L_1 phases when so prepared.

The invention will be illustrated by the following examples:

Example 1

The following ingredients were mixed at 60°C and cooled to form a ringing gel:

Component	Solids (%)	w/w (%)
MINERAL OIL (100%)	20	20
"EMPICOL"® 0251/70J (70%)	11.2	16
"EMPIGEN"® BB (30%)	4.8	16
"GLUCAPON"® 215 CS UP (65%)	6	9.2
"EMPILAN"® KB2 (100%)	7.5	7.5
SODIUM CHLORIDE (100%)	2	2
PERFUME (100%)	0.5	0.5
ETHYLENE DIAMINE TETRACETIC ACID (100%)	0.1	0.1
CITRIC ACID (100%)	0.2	0.2
BENZOIC ACID (100%)	0.3	0.3
SODIUM HYDROXIDE (47%)	0.1	0.2
WATER	---	Balance

The method of mixing comprised the following steps:-

1. Charge 50% of water
2. Heat to 60°C
3. Add EDTA, sodium benzoate, citric acid and 47% NaOH dissolve with stirring
4. Add "EMPIGEN" BB
5. Add mineral oil and disperse with stirring
6. Add "EMPILAN" KB 2 and mix thoroughly
7. Add "EMPICOL" 0251/70j
8. Add remaining water
9. Add "GLUCAPON" 215 CS UP
10. Add further KB 2 until clear
11. Cool
12. Add evaporated water
13. Adjust pH

Physical Data

pH (10%)	: 5.5 ± 0.1	Density @ 20°C	: 1.0 ± 0.1 g cm^{-3}
Solids (%)	: ~ 53% (typical)	Appearance	: Clear or Hazy Gel
Odour	: Characteristic	Set Point (typical)	: 30°C
Viscosity @ 20°C	: N/A		

The product was examined by x-ray diffraction and exhibited peaks at 13.145nm (intense and sharp), 7.943nm (ill defined) and 6.355nm (small), indicating cubic symmetry, and formed a clear microemulsion on dilution or heating. The latter gave good even distribution of oil applied to skin.

Example 2

The following ingredients were mixed at 60°C and cooled to form a ringing gel:

Component	Solids (%)	w/w (%)
MINERAL OIL (100%)	15	15
"EMPICOL"® CDL30J/35 (22%)	8	35.4
"EMPIGEN"® BB (30%)	8	26.7
"EMPICOL"® 0785 (40%)	2	5
"EMPILAN"® KB2 (100%)	6	6
"EMPILAN"® KB6 (100%)	6	6
CITRIC ACID (100%)	0.5	0.5
PERFUME (100%)	0.2	0.2
ETHYLENE DIAMINE TETRACETIC ACID (100%)	0.2	0.2
"KATHON"®	---	0.2
WATER	---	Balance
TOTAL	**45.8**	**100**

Physical Data

Appearance	: Clear Liquid/Gel	Odour	: Characteristic Odour
Solids	: 36.5% (typical)	pH (100%)	: 5.5 - 6.5 (typical
Odour	: Characteristic	Set Point	: 20 ± 5°C

Viscosity (Carrimed Rheometer @ 20°C : N/A

The product had small angle x-ray diffraction peaks characteristic of cubic symmetry and formed a clear microemulsion on dilution with water or warming. The latter gave good even deposition of oil on skin.

Examples 3 and 4

The following ingredients were mixed at 60°C and cooled to form ringing gels:

Component	1		2	
	Solids (%)	**w/w (%)**	**Solids (%)**	**w/w (%)**
"EMPIGEN"® CDL30J/35 (22%)	8	36.4	8	36.4
"EMPIGEN"® BB (30%)	8	26.7	8	26.7
"EMPICOL"® LB40 (40%)	4	7.5	3	7.5
"EMPICOL"® CVH (90%)	4	4	---	---
"EMPILAN"® KB2 (100%)	5.5	5.5	6	6
TRIETHANOLAMINE (100%)	1.1	1.1	---	---
CITRIC ACID	1	0.75	0.75	0.75
ETHYLENE DIAMINE TETRACETIC ACID	0.05	0.05	0.05	0.05
"KATHON"® CG (100%)	0.05	0.05	0.05	0.05
LIGHT MINERAL (100%)	14	14	20	20
WATER	---	Balance	---	Balance
TOTAL	**45.7**	**100**	**46.1**	**100**
Appearance	Clear Gel		Clear Gel	

The products in each case exhibited cubic symmetry and formed clear microemulsions or dilution with water or heating. The registered trade marks noted above have the following significance:-

"EMPICOL" CVH is a C_8 alkyl ether carboxylic acid
"EMPICOL" LB40 is a C_8 C_{10} alkyl sulphate
"EMPICOL" 0251/70J is a C_{12-14} alkyl 3 mole ethoxy sulphate
"EMPICOL" 9758 is a C_{10} alkyl sulphate
"EMPIGEN" BB is a C_{12-14} alkyl betaine
"EMPIGEN" CDL is coconut ampho acetate
"EMPILAN" KB2 is a C_{12-14} alkyl 2 mole ethoxylate
"EMPILAN" KB6 is a C_{12-14} alkyl 6 mole ethoxylate
"GLUCAPON" 215CS is a C_{8-10} alkyl polyclucoside D.P. 1.5
"KATHON" CG is a proprietary biocide